APR 2 1965

GENERAL ATOMIC

DIVISION OF **GENERAL DYNAMICS**

AEC RESEARCH AND
DEVELOPMENT REPORT

GA-6163
MASTER

A PROGRAM OF BASIC RESEARCH ON MECHANICAL
PROPERTIES OF REACTOR MATERIALS

QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING JANUARY 31, 1965

Prepared under
Contract AT(04-3)-167
Project Agreement No. 4
for the
San Francisco Operations Office
U.S. Atomic Energy Commission

February 25, 1965

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GENERAL ATOMIC
DIVISION OF
GENERAL DYNAMICS

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PREVIOUS REPORTS IN THIS SERIES

GA-5340—November, December, 1963; January, 1964

GA-5520—February, March, April, 1964

GA-5620—May, June, July, 1964

GA-5734—Summary report of work from November 1,
1963, through October 31, 1964

I. DEFORMATION MECHANISMS IN BCC METALS

INTRODUCTION

During this quarter major emphasis was placed on obtaining good measurements by means of the potential-drop resistivity method of the purity of the single crystal tungsten being produced. At impurity content levels of less than about 1 ppm, most chemical and spectrographic techniques become useless. The work of Seraphim, *et al.*,⁽¹⁾ allows one to relate the Γ , or residual resistivity, of a sample at 4.2°K to the total impurity content by the relation $c \sim 2.7 (1/\Gamma)$. However, as defects in the crystal are also detected by this method, other means must be utilized to ensure freedom from defects. The crystals produced by electron-beam floating-zone refining are probably free from defects because of the large length to radius ratio, slow growth rate, and lack of restraint during cooling. X-ray diffraction photographs have indicated freedom from defects such as sub-boundaries by the sharpness of the Laue spots (sub-boundary angles as small as 30' can be detected in this way).

RESISTIVITY MEASUREMENTS

By measuring the potential drop along a specimen with a known current passing through it, the relative resistance can be determined at different temperatures. The usual practice is to relate the resistance at a temperature where thermal lattice vibrations cause a significant contribution (conveniently chosen as room temperature) to the resistance at a temperature low enough to reduce electron-phonon interaction to negligible levels. The boiling point of liquid helium (4.2°K) is the lowest temperature which can be reached with reasonable ease and economy. The resistance at a sufficiently low temperature will be due to several factors, as explained in the section titled "Calculation of Factors Influencing the Resistivity Ratio"; however, the only factors subject to external change are specimen diameter, interstitial or substitutional impurity content, and lattice defects. The specimen diameter and defects can be controlled so that observed differences can be related to impurity content.

EXPERIMENTAL TECHNIQUE

Typical specimens are about 2 mm in diameter and 6 to 15 cm long, allowing the specimen and a suitable mounting jig to be immersed in a

liquid helium storage dewar which has a 15.8-mm neck. This eliminates the loss of liquid helium during transfer to a special dewar for testing. Stainless steel and Micarta or Teflon have many desirable properties for use in liquid helium and can be readily machined to the configuration required. Figure 1 is a drawing of the jig showing the wiring connections. Contact with the specimen is made by spot welding 0.075 mm (0.003 in.) diam platinum wires to the specimen. These wires are first soldered to pins in the Micarta to which are also soldered the lead wires to the switch box. Heavy-current lead wires are attached to the specimen at its bottom end by a Be-Cu spring clip and at the top by soldering them to a piece of nickel strip previously spot welded to the specimen.

The current is supplied from a storage battery at 2, 4, or 6 v and maintained constant at a given test temperature by a ballast resistor which can be adjusted to maintain the same current at various specimen temperatures (Fig. 2). The current is measured with a precision shunt. The generation of thermal emf's as much as 10 to 100 times the potential to be measured is the major problem of the tests. Low-thermal-emf-producing components are used throughout and the heat-producing ballast resistors are kept well away from the rest of the system. A current reversing switch is used to change the direction of current flow in the specimen so that the thermal emf's can be eliminated from the data reduction by subtracting pairs of readings and dividing the result by 2. For example,

$$\frac{(+2\mu v) - (-40\mu v)}{2} = \frac{42}{2} = 21\mu v .$$

Figure 3 shows the box containing the shunt, reversing switch, and two Leeds and Northrup switches for connecting any pair of potential probes to the cable leading to the Keithley 148 Nanovoltmeter. This apparatus and the Nanovoltmeter appear capable of measuring Γ of over 150,000. This was determined on a low- Γ specimen at a lower current level that gave potential drops of the order of those which would be observed on a specimen with a Γ of $\sim 150,000$.

ANNEALING EXPERIMENTS

The tungsten single crystal being used in the following resistivity experiments (specimen TH-15) was deformed by rolling at a temperature of $\sim 150^{\circ}\text{C}$. The crystal was rolled to about a 12% reduction of area (RA), heated in air to $\sim 1000^{\circ}\text{C}$, and then rolled to a further 12% RA. The resistivity ratio of this crystal changed from $\Gamma = 54,000$ before deformation to $\Gamma \sim 180$ following rolling. With the improved technique for gamma measurements described above, an examination of annealing of cold-work

TOP VIEW, BOTTOM END

BOTTOM VIEW, BOTTOM END

Fig. 1--Fixture for potential drop resistivity measurements in liquid helium

Fig. 2--Ballast resistors and battery for resistivity jig

Fig. 3--Selector switch and reversing switch box for resistivity jig

damage in a tungsten single crystal was started. The material chosen was the warm-rolled crystal described above. The Γ was determined in the manner previously described. The sample was annealed for 30 min at a series of increasing temperatures and Γ determined between each anneal.

Liquid baths were planned for the annealing experiment to ensure the rapid rise to temperature of a specimen placed in such a bath. Also, it was hoped that by excluding air the contamination of the specimen could be minimized and surface effects interfering with welding of the potential leads could be prevented. Boiling water (100°C) was used for the first temperature bath. For the temperature range up to 300°C diglycol laurate was chosen as the bath liquid; however, it tended to break down when heated for extended periods and was subsequently used only to 260°C . For the next range of temperatures, a $\text{NaNO}_2\text{-KNO}_3$ salt bath was used. Below about 350°C the salt has little effect on the tungsten; however, at 385°C the bath removed about 0.06 mm from the surface of the crystal during the anneal, and some crystallographic etching seemed to be occurring. The Γ decreased about 2% also. Lead baths are in use above 350°C and will be used up to about 1000°C . Some lead and dross adheres to the specimen on removal, but a 30-sec dip at 335°C in the salt bath used previously cleans the specimen.

It was decided to perform resistivity studies on deformed tungsten for several reasons: first, to evaluate and debug the equipment which had been constructed for this purpose; second, to determine suitable methods of annealing the specimens without introducing impurities; and third, to obtain isochronal annealing curves for deformed tungsten which could serve as a basis of comparison for the irradiated crystals. Details of the measurements are contained in Table 1. The measurements are presented in the form of resistivity ratios, Γ , defined as the ratio of resistance at 273°K to that at 4.2°K . This quantity is more convenient to use than the absolute resistance, since by using the ratio of the measurements at the two temperatures, the shape factor, which must be considered in determining the absolute resistivity, cancels for all practical purposes. This point has been discussed by Kunzler and Wernick.⁽²⁾

Since measurements at 77°K had been considered for this work, it was also, in part, the object of these first experiments to evaluate and compare results at this temperature with those obtained at 4.2°K . The 77°K measurements are also presented in Table 1. The measurements have been extended to 543°C ; however, the scatter in results at 77°K compared with that at 4.2°K is obvious. Since the newly constructed apparatus conserves helium, all future measurements will be performed at 4.2°K . Measurements made at 4.2°K , of course, also have the added advantage that scattering of conduction electrons by thermal phonons is reduced to low levels. The difference may be seen by comparing $\Gamma_{770\text{K}}$

Table 1
 ISOCHRONAL ANNEAL OF ROLLED SINGLE
 CRYSTAL TUNGSTEN (TH-15)

(Current 1 amp; probe spacing ~ 4.5 cm)

Conditions	Γ_{77}	$\Gamma_{4.2}$	$\rho_{4.2}^* \times 10^3$
As-grown ^a	10.0	$\sim 54,000$	0.02
Rolled $\sim 20\%$	9.0	182	5.52
30 min anneal at			
100°C	8.65	181	5.56
230	9.68	185	5.44
275	9.10	186	5.40
308	----	187	5.38
336	8.92	187	5.38
384	9.10	176	5.72
382	8.9	177	5.68
425	9.2	201	5.00
454	9.01	227	4.43
473	9.13	249	4.03
505	9.35	269	3.74
543	9.23	272	3.70

^aCurrent 6 amp.

with $\Gamma_{4.2^\circ\text{K}}$ in Table 1. Measurements of the annealing-out of defects with temperature are usually presented in a dimensionless form such as

$$\rho^* = \frac{R_{4.2^\circ\text{K}}}{R_{273^\circ\text{K}} - R_{4.2^\circ\text{K}}}, \quad (1)$$

where $R_{4.2^\circ\text{K}}$ is the absolute resistivity at 4.2°K , and $R_{273^\circ\text{K}}$ is the absolute resistivity at 273°K . However, since our measurements are in the form of resistivity ratios, $\Gamma = R_{273^\circ\text{K}}/R_{4.2^\circ\text{K}}$, Eq. (1) can be simply transposed to

$$\rho^* = \frac{1}{(\Gamma - 1)}. \quad (2)$$

This quantity has been given in Table 1 and is also plotted versus annealing temperature in Fig. 4. The anneal at 384°C in the salt bath resulted in pitting of the specimen and an increase in ρ^* . The increase in ρ^* may well be associated with the pickup of foreign matter. This will not be known until the sample has been annealed at very high temperatures ($\sim 2500^\circ\text{C}$), which should restore the original measured Γ of $\sim 54,000$. As previously discussed, measurements above 384°C have been performed in a lead bath and the quantity ρ^* can be seen to have decreased in the expected fashion. The temperature range of the recovery for this specimen (400° to 500°C) appears to be about the same as that previously reported by Schultz⁽³⁾ for cold-worked tungsten. This reported stage III recovery appeared to be completed at $\sim 475^\circ\text{C}$. The annealing rates ($\Delta T/t$) for the two cases were similar (Schultz⁽³⁾ ~ 1.3 , and this work ~ 0.83).

A point of considerable interest, and one briefly covered in the last annual summary report, GA-5734,⁽⁴⁾ was the influence of variables such as scattering of conduction electrons at the surface on the measured values of Γ . This is known to be extremely important for small-diameter samples and especially so for samples of high purity. The following calculations were performed in order to obtain a more realistic value for Γ , and hence impurity content.

CALCULATION OF FACTORS INFLUENCING THE RESISTIVITY RATIO

The resistance ratio, $r(T) = \rho(T)/\rho(273^\circ\text{K})$, of the electron-beam zone-refined tungsten single crystal used in the rolling experiment (specimen TH-15) was measured at liquid helium temperature. The experimental sample was approximately a cylinder with a diameter of 0.216 cm , and the experimental current was approximately 6 A ; the result is $r(4.2^\circ\text{K}) = 1/54,000$. The question is: To what extent is the observed electrical

Fig. 4--Plot of isochronal annealing curve for deformed tungsten single crystal TH-15; annealing time, 30 min

resistivity determined by

- a. Atomic imperfections and other mechanical defects such as dislocations;
- b. Orbital scattering of conduction electrons at displaced atoms and on impurities at the surface of the crystal;
- c. Scattering of conduction electrons by thermal phonons;
- d. Coulomb scattering of conduction electrons by one another?

In order to find, from the observed resistivity value at 4.2°K , the residual resistivity of the bulk material, ρ_0 , which is a measure for the impurity of the material and for the mechanical defects in the lattice, one must evaluate (b), (c), and (d) above. To this end let us write the total resistivity of a transition metal in the form

$$\rho(T) = \rho_0 + \rho_1 + \rho_{\text{ph}} + \rho_{\text{s-d}} + \Delta\rho_1, \quad (3)$$

where ρ_0 and ρ_1 are temperature independent and correspond to (a) and (b), respectively, and where ρ_{ph} and $\rho_{\text{s-d}}$ are temperature dependent and correspond to (c) and (d), respectively. The last term, $\Delta\rho_1$ is the temperature-dependent deviation from Matthiessen's rule. We shall discuss the different contributions to ρ in terms of a simple model for the electronic structure of a 5d transition metal, namely, Mott's isotropic two-band model.⁽⁵⁾ It presumes that for all directions in momentum space, the Fermi surface cuts the same 6s- and 5d-like branch of the conduction or valence bands. Strictly speaking, this is not the case, as one knows from the branch-structure calculations of Manning and Chodorow.⁽⁶⁾ Since, however, the electrical resistivity in a zero magnetic field is an isotropic tensor, the simple two-band model works quite well for this transport coefficient. Explicit formulas for ρ_{ph} and $\rho_{\text{s-d}}$ can be found in the literature.⁽⁷⁾⁽⁸⁾ The third temperature-dependent part of ρ , the deviation $\Delta\rho_1 (> 0)$ from Matthiessen's rule, has also been calculated for the two-band model.⁽⁹⁾

First, let us assess the temperature-dependent resistivities at 4.2°K . It is readily seen that

$$\rho_{\text{s-ph}} \ll \rho_{\text{s-d}} \text{ at } 4.2^{\circ}\text{K},$$

or, correspondingly, that the mean free path of s-electrons due to phonon scattering, $\ell_{\text{s-ph}}$, is much greater than $\ell_{\text{s-d}}$, where $\ell_{\text{s-d}}$ is the mean free path of s-electrons due to Coulomb scattering by d-electrons. The same

holds for d-electrons. With

$$\frac{1}{\rho_{s-ph}} = \frac{n_s e^2 \ell_{s-ph}}{m_s^* v_{s\zeta}} = A T^{-5} \quad (T \ll \theta_D = 315^\circ K), \quad (4)$$

where n_s is the concentration of s-electrons and m_s and $v_{s\zeta}$ are their effective mass and Fermi velocity, respectively, we have

$$\ell_{s-ph} = \frac{1.82 \times 10^4}{T^5} \text{ cm} \quad (\text{with } T \text{ in } ^\circ K), \quad (5)$$

provided we take the following parameter values:

$$\left. \begin{aligned} A (\text{exp})^\dagger &= 1.63 \times 10^{15} \Omega^{-1} ^\circ K^5, \\ n_s &= 3.2 \times 10^{22} \text{ cm}^{-3},^{\dagger\dagger} \\ v_{s\zeta} &= 10^8 \text{ cm/sec}, \\ m_s &= m (\text{electron rest mass}). \end{aligned} \right\} \quad (6)$$

The mean free path of s-electrons due to Coulomb scattering by d-electrons is given by⁽⁸⁾

$$\frac{1}{\ell_{s-d}} = \frac{1}{n_d Q_{sd}} \frac{v_{rel}}{v_{s\zeta}} \frac{(kT)^2}{\zeta(\zeta - A)}, \quad (7)$$

where n_d is the concentration of d-electrons, Q is a collision cross section of the order of the square of the screening radius, v_{rel} is an average relative velocity for electrons at the Fermi surface, and A is an energy which measures the overlap of the s- and d-bands. The T^2 dependence of ρ_{s-d} corresponding to Eq. (7) has also been observed by Berthel;⁽¹⁰⁾ its physical origin was, however, not recognized. With

$$\frac{1}{\rho_{s-d}} = \frac{B}{T^2}, \quad (8)$$

[†] See Ref. 10. The author has observed a T^5 dependence of $\rho_{ph} \simeq \rho_{s-ph}$ for $18^\circ K \lesssim T \lesssim 30^\circ K$. At $20^\circ K$, $\rho_{s-ph}/\rho(273) = 3.7 \times 10^{-4}$. With $\rho(273) = 5.3 \times 10^{-6}$ cm one has the above value for A .

^{††} It is assumed that $n_s = n_d = 1/2$ times the number of atoms per cm^3 . With a lattice constant $a = 3.15 \times 10^{-8}$ cm, and with 2 atoms per unit cell (bcc) one is led to the above value of n_s .

one finds from Berthel's data that $B_{\text{exp}} = 1.09 \times 10^{12} \Omega^{-1} \text{ cm}^{-1} \text{ } ^\circ\text{K}^2$. The T-dependence of the corresponding free path is given by

$$\ell_{\text{s-d}} = \frac{24}{T} \text{ cm} , \quad (9)$$

with T in $^\circ\text{K}$. At 4.2°K one has $\ell_{\text{s-ph}} \simeq 10\ell_{\text{s-d}}$. As for the third temperature-dependent contribution to ρ , $\Delta\rho_{01}(T)$, it has been discussed, together with experimental data on cold-worked tungsten, in great detail by Krautz and Schultz.⁽¹¹⁾ From their work it can be seen that our single crystal, TH-15, is sufficiently pure to justify the approximate relation given by

$$\Delta\rho_{01}(T) = \gamma(\rho_{\text{s-ph}} + \rho_{\text{s-d}}) , \quad (10)$$

where

$$\gamma = \frac{1}{\mu} \left(\frac{\lambda - \mu}{1 + \lambda} \right)^2$$

with

$$\lambda = \frac{\rho_{\text{s}}(0)}{\rho_{\text{d}}(0)} , \quad \mu = \frac{\rho_{\text{s-ph}} + \rho_{\text{s-d}}}{\rho_{\text{d-ph}} + \rho_{\text{d-s}}} .$$

Krautz and Schultz find that γ depends only slightly on the residual resistivity $\rho_0 = \rho_{\text{s}}(0) + \rho_{\text{d}}(0)$; for their best sample $\gamma = 1.13$. If we take $\gamma = 1$, the total temperature-dependent resistivity at low temperature is given by Eqs. (4), (8), and (10):

$$\rho_{\text{s-ph}} + \rho_{\text{s-d}} + \Delta\rho_{01} \simeq \frac{2}{B} T^2 . \quad (11)$$

At $T = 4.2^\circ\text{K}$ the right-hand side is equal to $3.22 \times 10^{-11} \Omega \text{ cm}$.

The temperature-independent part of ρ is found by subtracting the resistivity value given by Eq. (11) from $\rho_{\text{exp}}(4.2^\circ\text{K}) = \rho(273)/54,000 = 0.98 \times 10^{-10} \Omega \text{ cm}$:

$$\rho_0 + \rho_1 = 6.6 \times 10^{-11} \Omega \text{ cm} . \quad (12)$$

In order to find ρ_0 , the resistivity due to surface scattering, ρ_1 , must be estimated. The experimental sample has approximately the geometrical form of a thin wire with a radius of 0.108 cm. The surface resistivity of a thin wire has been evaluated by Nordheim⁽¹²⁾ and by Dingle.⁽¹³⁾ The

pertinent parameter is

$$k = 2r/\ell_0, \quad (13)$$

where ℓ_0 is the free path corresponding to ρ_0 --i.e., the residual resistivity of the bulk material. ρ_0 is given by

$$\frac{1}{\rho_0} = \frac{n_s e^2 \ell_0}{m_s^* v_{s\zeta}} = \frac{1}{\rho_{s0}} + \frac{1}{\rho_{d0}}, \quad (14)$$

where, by definition,

$$\ell_0 = \ell_{s0} \left(1 + \frac{m_s^* v_{s\zeta} \ell_{d0}}{m_d^* v_{d\zeta} \ell_{s0}} \right). \quad (15)$$

If one knows $\rho_1(k)$, then Eq. (12), in principle, determines ℓ_0 . In order to find ℓ_0 experimentally, one measures, at a given temperature, the residual resistivities of wires with different diameters. Without such data we can roughly estimate ρ_1 with the help of tabulated values for the surface resistivity, which are found in Dingle's paper. His formula (16.3) reads

$$\frac{\rho_0}{\rho_1 + \rho_0} = (1 - \epsilon)^2 \sum_{n=1}^{\infty} n \epsilon^{n-1} \left[\frac{\rho_0}{\rho_1 + \rho_0} (nk) \right]_{\epsilon=0}, \quad (16)$$

where $\epsilon = 0$ for diffuse and $\epsilon = 1$ for specular surface scattering. Since $\ell_0 > 0.165$ cm (Eq. (12)), one has $k < 1.3$. Assuming that $k = 1.3$ and that $\epsilon = 0$, one finds with Dingle's numerical data the following result:

$$\frac{\rho_0}{\rho_1 + \rho_0} = 0.55. \quad (17)$$

Hence, 45% of the residual resistivity at $T = 0$ is caused by surface scattering. For the tungsten single crystal the residual resistivity of the bulk material is given by ρ_0 (TH-15) = $3.6 \times 10^{-11} \Omega \cdot \text{cm}$. The corresponding value of $\rho(273)/\rho_0 = 147,000$. This number should be reliable within $\pm 20\%$.

If one now assumes the corrected value of $\Gamma = 147,000$ to be a measure of electrons scattered by impurities, and mechanical defects to be a measure of the corresponding concentrations of impurities, the relationship

$$C \leq 2.7 \frac{R_{4.2^{\circ}\text{K}}}{R_{273^{\circ}\text{K}}} \text{ atom-\%} , \quad (18)$$

yields $C \sim 1.8 \times 10^{-7}$ atom-%. This is sufficiently low to permit the study of neutron damage per se.

MECHANICAL PROPERTY MEASUREMENTS AND IRRADIATIONS

The studies of irradiated tungsten crystals include measurements of the changes in mechanical properties due to irradiation and their recovery with annealing.

The proportional limit is known to be sensitive to specimen alignment. For this reason, during the last quarter universal knife edges have been designed and constructed to ensure axial alignment of the specimens during testing. Figure 5 presents drawings of the designed unit, and Fig. 6 shows a specimen installed in the grips with the strain-gauge extensometer in place.

Evaluation is presently in progress. Two tests have been performed on tungsten crystals which had been welded into stainless steel grips; however, these crystals fractured at the welds at low stresses. Examination revealed that a reaction of some type had occurred between the tungsten and the stainless steel. The use of stainless steel end grips has been discontinued.

Zone refining of tungsten crystals is continuing and new irradiation capsules are being planned to cover additional dose ranges.

The first irradiation capsule⁽⁴⁾ has been discharged from LITR and should be received at General Atomic shortly. The integrated dose based on reactor operating history was 1.6×10^{19} nvt. The capsule contains monitor wires of iron and titanium, and they will be counted on receipt in order to independently determine the dose.

Fig. 5--Universal knife edges

Fig. 6--Universal knife edge joints with specimen and extensometer

II. DISLOCATION DYNAMICS

INTRODUCTION

A new method of measurement of mechanical-strain relaxation is described and applied to the problem of determining the stress-dislocation strain curve of a bcc transition metal--Nb. The method enables the dislocation-relaxation spectrum of a specimen to be measured over a wide range of temperatures and, hence, relaxation times. Perhaps the most important feature of this method is that it enables the dislocation strain component to be completely extracted from the total observed strain, even for very small (micro) strains. Thus, for the first time a true stress-dislocation strain curve can be measured. It is shown by this method that there exist several "micro-yield" points prior to the usual macro-yield point. Finally, this method is used on several other bcc transition metals (W, Fe, Ta) to show the similarities in the relaxation structures of these metals.

In all of the bcc transition metals there exist two main dislocation relaxations, a low-temperature relaxation which will be referred to as the L relaxation, and a high-temperature relaxation or H relaxation. The α and β relaxations discovered previously by internal friction methods⁽¹⁴⁾ are shown to be the fine structure of the L relaxation, whereas the γ relaxation is identified with the H relaxation. The total amount of dislocation strain contributed by the L relaxation is shown to be limited or restricted to about 1 to 5×10^{-4} , depending on the impurity concentration and previous mechanical history. The H relaxation is shown to control gross plastic flow near room temperature. The practical aspects of these relaxations are discussed in terms of their relevance to the ductile-brittle transition problems.

THE CONSTANT-TEMPERATURE-RISE STRAIN RELAXATION (CTRSR) METHOD

This method of making microstrain measurements was first described by R. H. Chambers in an invited paper at the February, 1964, AIME Meeting in New York.⁽¹⁵⁾

The method consists of first taking a specimen to as low a temperature as possible (4.2° or 50° K), then loading the specimen to a given

stress. (In all the measurements discussed below the torsional-deformation mode is used, but the method is, in principle, applicable to any deformation mode.) After loading, the specimen is heated at a constant temperature rate and measurements are made of the resulting deformation as a function of time (and hence, of temperature). The curve of strain versus temperature which results from these measurements will be called a "stressed-strain release" (S-SR) curve. If, instead of strain, a quantity like energy were being measured, the S-SR curve would resemble a stored-energy release curve. In Fig. 7 is shown a schematic S-SR curve; at several temperatures there exist "steps" in the strain--corresponding to a particular strain release. The most important features associated with the S-SR curves are the average temperatures of the releases, the breadth in temperature of a particular release, and the total amount of strain in each release. A further important variable is the size of the initial applied stress. In Fig. 8 is shown a composite of a number of CTRSR runs in Nb (specimen TF-2), where each S-SR curve was made with a different applied stress. It is important to note that the specimen measured in Fig. 8 had received a prestrain of $\sim 20\%$ and that the total strains accrued during the CTRSR runs did not exceed 1% . As a result of this large prestrain, reruns of S-SR curves at various stresses were reproducible.

Figure 8 shows that as the applied stress is increased, the total amount of strain released increases, as would be expected. The major strain releases can be seen to be located in this Nb specimen near 100°K (the L release) and 230°K (the H release). The L release is very broad, extending from 50°K up to nearly 150°K . On the other hand, the H release is relatively narrow, starting near 210°K and extending up to 250°K . Another important feature is shown in Fig. 8. There is a threshold stress, τ_c , which if not exceeded will not produce large amounts of plastic flow, even at higher temperatures. In the specimen shown in Fig. 8, this τ_c is $\sim 13 \times 10^{-4} \text{ G}$, where G is the shear modulus of Nb. When stresses in excess of τ_c are applied at low temperature, large amounts of plastic flow begin on reaching the H-release temperature. Furthermore, it can be seen that the greater the applied stress, the lower the temperature becomes where large amounts of plastic flow occur. Also, careful examination of the L-release region shows that the amount of strain produced in this region does not increase as fast as the applied stress. Thus, the mechanical behaviors of the L and H relaxations are markedly different from one another.

In Fig. 9 is shown another type of CTRSR run designed to separate the elastic and dislocation components of strain. Curve "a" was derived as described above, and for a particular applied stress, it gives the total strain accrued throughout the temperature interval 50° to 300°K . Curve "b" was obtained following curve "a" when on reaching 300°K the temperature was lowered quickly to the original starting temperature (50°K),

Fig. 7--Schematic of the CTRSR method

Fig. 8--CTRSR runs at various stresses for Nb-TF-2; prestrained 20% at room temperature

Fig. 9--CTRSR runs for Nb-TF-2; curve "a," regular run; curve "b," second temperature run

while still maintaining the same stress used to obtain curve "a." Following this reduction in temperature to 50°K, the mechanical zero was adjusted to agree with the starting point used for curve "a" and then the temperature was increased again at the same fixed rate as for curve "a." The resulting curve of deflection versus temperature is curve "b." If this last procedure is repeated several times, curve "b" is still obtained. Thus, the curve-"b" state is completely reversible. Measurements of the slope of strain versus temperature of curve "b" agree well with measurements of the temperature dependence of the shear modulus of Nb; thus, curve "b" represents the strictly elastic component of strain. By subtracting curve "b" from curve "a," the temperature dependence of the dislocation strain may be obtained. Also shown in Fig. 9 are "a" and "b" curves measured at higher stresses. As can be seen from these measurements, when the applied stress exceeds a certain critical value, τ_c , the "b"-type measurement shows evidence of plastic deformation when the temperature becomes high enough. At these temperatures curves "a" and "b" tend to join. It is clear that the "b"-type measurements represent a kind of steady-state type of "creep" in which the strain seen in the "a"-type measurement has been exhausted. Thus, one would expect a "b"-type curve to obey the thermally activated equation

$$\dot{\epsilon}_d = \dot{\epsilon}_0 \exp \left[- \frac{H^*(\sigma, T)}{kT} \right], \quad (19)$$

where $\dot{\epsilon}_d$ is the dislocation strain rate determined using the method described above and the relation

$$\dot{\epsilon}_d = \frac{d\epsilon_d}{dt} = \frac{d\epsilon_d}{dT} \times \frac{dT}{dt}, \quad (20)$$

where dT/dt is the constant heating rate used (0.03°K sec⁻¹). Plots of $\ln \dot{\epsilon}_d$ versus $1/T$ obtained from data in Fig. 9 show very good straight lines over temperature intervals of 40° to 50°K near 250°K. The activation energies obtained show (1) dependence on the applied stress that is consistent with the stress-assisted nature of the thermally activated processes under study, and (2) that the limiting energy H_0 of the process active near 230°K is ~0.8 ev. Thus, these measurements have been able to provide values of the average activation energy of the H process as well as to isolate the strictly dislocation strain component.

Returning again to Fig. 8, it is of interest to plot the isotherms derivable from this plot. Thus, if 100°K is chosen, one can obtain from Fig. 8 the stress versus microstrain curve for that temperature. In Fig. 10 is plotted a series of these isotherms starting from 80°K and extending up to ~300°K. Since all of these data were taken at a constant

Fig. 10--Isothermal stress-dislocation strain curves derived from data in Fig. 8

heating rate, dT/dt , and $d\epsilon_d/dt = d\epsilon_d/dT \times dT/dt$, then the dislocation-strain rate associated with these stress-microstrain curves is proportional to the slope $d\epsilon_d/dT$ on Fig. 8. Since $d\epsilon_d/dT$ remains bounded to within about one order of magnitude, the dislocation-strain rate can be considered to be known within one order of magnitude. For the curves in Fig. 10, the average dislocation strain rate is $\sim 10^{-7} \text{ sec}^{-1}$.

Several important features should be noted in Fig. 10: At temperatures above $\sim 220^\circ \text{K}$, the stress-dislocation strain curves resemble the parabolic flow behavior of a metal like aluminum; no yield point is observable. Below 200°K , there develops an apparent "hardening" which increases as the temperature is decreased. This hardening increases in magnitude so much that at 80°K it completely obscures the initial flow region. It is important to observe that although the stress-dislocation strain curves below 200°K show an increase in the stress necessary to produce large amounts of dislocation strain, this increase is reversible. Thus, if the stress at, say, 120°K is reduced to zero and reapplied in the opposite sense (right-hand twist versus left-hand twist), the same stress-dislocation strain curve is reproduced with the signs of the stress and strain reversed. If the source of the increase in stress (hardening) to produce plastic flow below 200°K were due to an increase in dislocation density or other mechanical state change which is essentially irreversible, then one would not expect to be able to reproduce the stress-dislocation strain curves. On the basis of the reproducibility of these curves, the explanation of the increased stress must lie in some essentially reversible mechanism, such as a nonlinear elastic response of dislocations.

It is proposed to explain the above phenomena in terms of the multi-component model of dislocation motion outlined previously.⁽¹⁴⁾ In terms of this model, L-type dislocations are restricted in their motions at low temperatures by barriers formed by H-type dislocations which are "frozen in" at these temperatures. The initial "easy flow" observed at low temperatures (below 200°K in the above experiment) is due to the movement of the L-type dislocations. These dislocations are exhausted as they run up against the H-barriers and thus contribute only a limited amount of plastic flow strain ($\sim 2 \times 10^{-4}$); the stress necessary to produce further plastic flow increases until the H-type dislocations can be stress-assisted to move at the low temperature. Until the H-type dislocations move, the movement of the L-type dislocations can be considered to take place under nonlinear elastic constraints.

The following two questions are related to the above discussion and are currently under investigation: (1) What is the nature of each of the major dislocation types (L and H)? (2) How general is the behavior described above?

In Fig. 11 is seen an answer to the second question: CTRSR runs were made on Ta, Nb, W, and Fe, and are plotted as $\log \epsilon$ versus T in Fig. 11 in order to accentuate the detail at small strains. It can be seen that all of these bcc transition metals exhibit L and H relaxations. Perhaps the most noteworthy effects are the very low temperatures of the L process in Fe compared with the other metals, and the relatively high temperature of the H process in W. It is believed that closer examination of these metals using the CTRSR technique would be useful in explaining their mechanical properties in terms of the multicomponent model.

At the present time the best estimate of the nature of the dislocations contributing to each process is that used in an earlier report⁽¹⁴⁾ to describe the α , β , and γ processes: α and β (L processes) are associated with edge and edge-screw dislocations, and γ (H processes) are associated with screw dislocations.

Fig. 11--CTRSR runs for Ta, Nb, W, and Fe

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